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# Combined effect of rapid nitriding and plastic deformation on the surface strength, toughness and wear resistance of steel 38CrMoAlA

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**Abstract.** The combined treatment of pressurized gas nitriding and cold rolling is proposed as a new approach to rapid preparation of a strong and tough nitrided layer for steel 38CrMoAlA. The microstructural characteristics and properties of the modified surface layer in comparison with those of the conventionally gas nitrided sample have systematically been evaluated. The results show that the hardness and toughness of the nitrided surface layer can be significantly improved by the combined treatment. Especially, the wear resistance of nitrided surface layer under heavy loads was greatly enhanced. It can provide a new approach to rapidly preparing a nitrided layer with high strength and toughness.

## 1. Introduction

Gas nitriding (GN), one of the well-established commercial surface modification techniques, has been widely used for improving the mechanical properties and wear/corrosion resistance of alloys and steels [1-3]. During GN, a gradient composite structure with a hard surface compound layer and a tough diffusion layer, is formed via thermochemical treatment in an NH<sub>3</sub> containing atmosphere. However, conventional GN processes are usually performed at 500~590°C for a long duration (20~80 h), which results in high energy consumption and low production efficiency in industrial applications [1-3]. Moreover, compared with the diffusion layer, the compound layer consisting of a heterogeneous mixture of  $\epsilon$ -Fe<sub>2.3</sub>N and  $\gamma'$ -Fe<sub>4</sub>N phase, is very thin and contains high internal stresses, so that the nitrided surface layer is friable and brittle [4-9]. This will affect the performance of the nitrided components subjected to severe service environments involving high shear, compressive and impact loading conditions [4,5,7].

Up to now, many rapid GN technologies have been developed to improve GN efficiency such as a surface nanocrystallization pretreatment [10,11], an activated GN by adding ammonia chloride [12,13], as well as a cathode sputtering pretreatment [14]. Meanwhile, some methods of nitriding such as two-stage GN and controlled GN with the nitriding potential below the critical value were



developed to obtain a single  $\gamma'$ -Fe<sub>4</sub>N phase or to form a nitrided surface without a compound layer [4,5,15]. However, it would take a longer duration when nitriding under the lower nitriding potential using an extremely low ammonia flow rate. Although the brittleness of the surface nitrided layer could be eliminated by means of the two-stage nitriding process, consisting of conventional nitriding and decomposition of the compound layer by ion sputtering, serious deterioration of the surface hardness and wear resistance of the nitrided steel will be induced [4]. Therefore, it is necessary to develop a technical method that can not only get a compound-free nitrided layer with excellent toughness on the steel surface, but also have a high production efficiency.

The previous works show that pressurized gas nitriding (PGN) can significantly accelerate the nitriding efficiency due to the faster reaction rates, the sufficient and stable nitrogen surface concentration under higher pressure [16,17]. It is noted that the nitriding potential of PGN can be adjusted by the nitriding pressure and ammonia flow rate. This implies that it is possible to rapidly prepare a compound-free nitrided layer by means of a PGN treatment using continuous NH<sub>3</sub>. In fact, it has been reported that the hardness of this tough nitrided layer can be further improved by means of cold rolling (CR) or a surface mechanical attrition treatment (SMAT) without leading to spalling on the steel surface [4]. But what effect of the combined treatment (PGN + CR) on the surface performance for the nitrided steel has not been clarified so far.

The present work aims to examine the effects of a combined treatment (PGN + CR) on the microstructure and surface properties of the nitrided steels, and compare with those in the CGN and PGN sample, and to provide theoretical and experimental warrants for developing a rapid preparation of a highly strengthened and toughened nitrided layer for steels.

## 2. Experimental procedure

The tested steel 38CrMoAlA was cut from a hot-rolled bar with the chemical composition (mass%) of 0.39 C, 1.62 Cr, 0.22 Mo, 1.04 Al, 0.31 Si, 0.42 Mn, 0.021 P, 0.018 S and bal. Fe. Before nitriding, the samples were oil quenched from 940°C, and tempered at 650°C for 1.5 h to produce the desired mechanical properties (with a hardness of 310 HV). All of the treated samples were cut to 60 mm × 50 mm × 3 mm, surface ground and washed carefully with acetone and absolute alcohol, then immediately hung in a pressurized GN furnace for nitriding [16]. For the nitriding treatment, high-purity NH<sub>3</sub> (99.99 vol.%) gas was used as the nitriding medium. The CGN was performed at 530°C for 30 h using a nitriding potential of  $r_N=1.96 \text{ atm}^{-1/2}$  (NH<sub>3</sub> flux: 0.5 l/minute) under 1 atm pressure, whereas the PGN was performed at 530°C for 5 h using a nitriding potential of  $r_N=0.26 \text{ atm}^{-1/2}$  (NH<sub>3</sub> flux: 0.2 l/minute) under 5 atm pressure. Nitriding potential is defined as  $r_N=p_{\text{NH}_3}/p_{\text{H}_2}^{3/2}$ , where  $p$  denotes partial pressure [1-3]. Note that, on applying this nitriding potential for PGN, hardly any iron nitrides can develop in the case of the nitrided sample, according to the Lehrer diagram [18]. After the PGN treatment, the PGN samples were cold rolled by 30%.

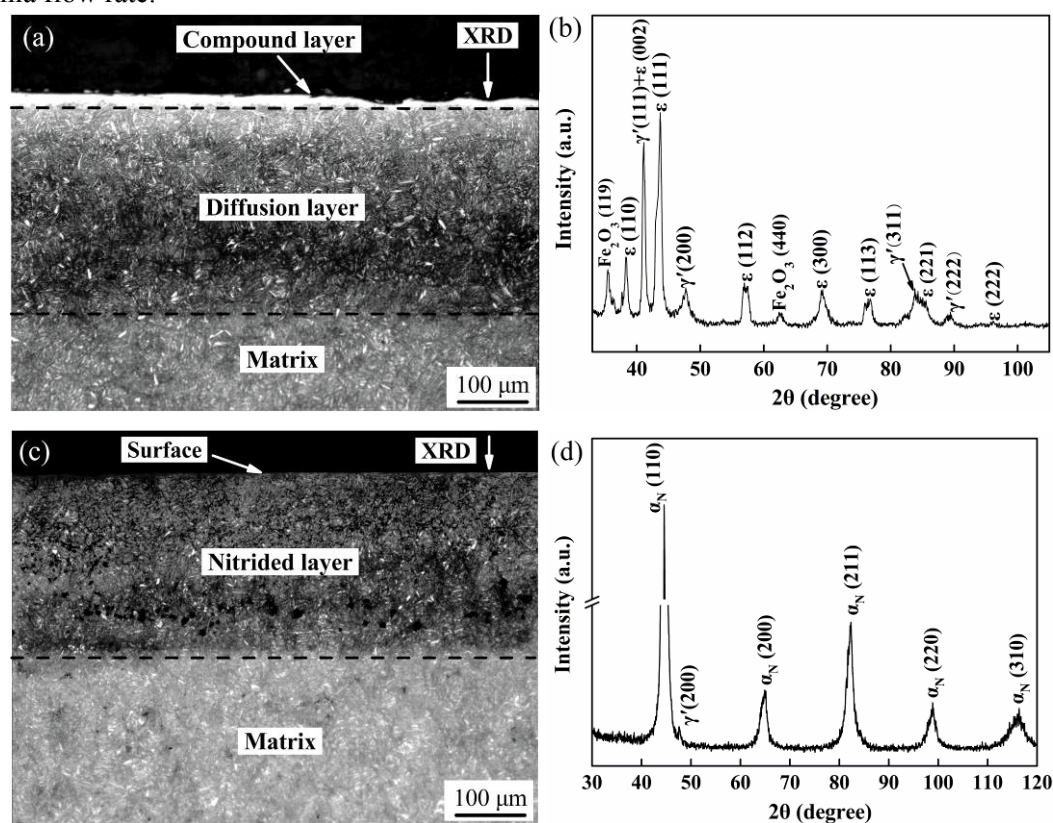
Cross-sectional observations of the nitrided samples were performed by optical microscopy (OM). The phases in the surface layer were identified by means of a D/MAX-PC 2500 X-ray diffractometer (XRD) using CuK $\alpha$  radiation, operated at 40 mA and 40 kV. A JEM-2010 TEM was employed to characterize the microstructural features in the surface layer of the PGN + CR (PGNC) sample. The depth-dependent hardness of the nitrided sample was measured using an FM-ARS 9000 micro-Vickers hardness tester with a load of 200 g and a holding time of 10 s. Before the toughness test, a mirror finish was performed to ensure minimal loss of surface layer, the surfaces of the treated samples were slightly polished using 1  $\mu\text{m}$  diamond paste. The polished surfaces were then subjected to a Vickers hardness test using a load of 10 kg.

The wear tests were carried out in a pin-on-disc machine at room temperature under 1 atm pressure. The GCr15 steel pin (with a hardness of 63~65 HRC) was turned in an anti-clockwise motion at 50 rpm in contact with the nitrided sample (disc) under two loads of 100 and 400 N, respectively. After a sliding distance of about 170 m, the experiment was stopped, and then the worn surfaces were examined using an S-4800 scanning electron microscope (SEM).

### 3. Results and discussion

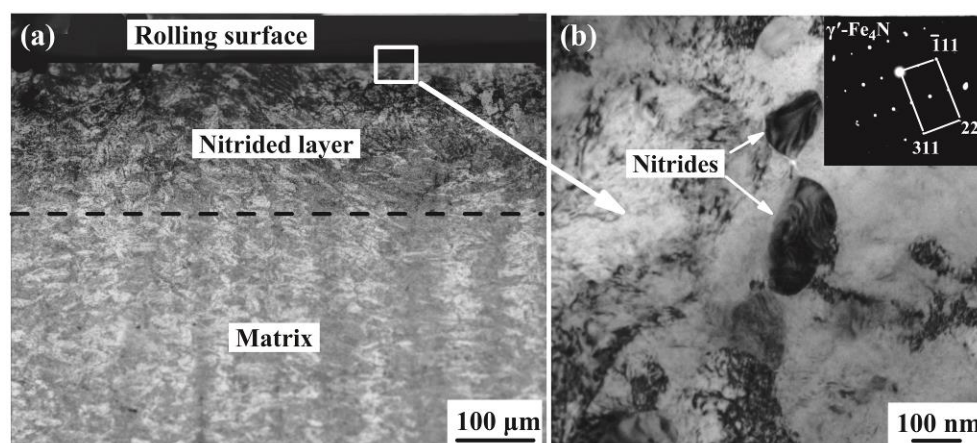
#### 3.1. Microstructural characterization of the surface layer

Fig. 1a shows the cross-sectional microstructure of the steel 38CrMoAlA sample after CGN at 530°C for 30 h. It was clearly observed that the nitrided layer was subdivided into a bright compound layer (CL) and a gray diffusion zone. The XRD patterns revealed that the CL with about 15  $\mu\text{m}$  in thickness was composed of  $\epsilon\text{-Fe}_{2.3}\text{N}$  and  $\gamma'\text{-Fe}_4\text{N}$ , as well as a few oxide phases (Fig. 1b). The presence of the oxide phases suggested that slight oxidation had occurred during the long GN duration. After the PGN treatment at 530°C under 5 atm for 5 h, the nitrided surface layer almost consisted of  $\alpha_{\text{N}}$  phases except for a very small amount of  $\gamma'\text{-Fe}_4\text{N}$  phase (Fig. 1d), which was consistent with the OM observation of the PGN sample (Fig. 1c). Moreover, it is worth noting that the thickness of the nitrided layer in the PGN sample is about 282  $\mu\text{m}$ , which is almost equal to that ( $\sim 316 \mu\text{m}$ ) after CGN for 30 h. The above results demonstrated that a compound-free nitrided layer can be rapidly prepared by PGN with a controlled nitriding potential, which was obtained through adjusting the nitriding pressure and ammonia flow rate.



**Figure 1.** Cross-sectional optical micrographic observations of (a) CGN sample and (c) PGN sample, and the XRD patterns of (b) CGN sample and (d) PGN sample.

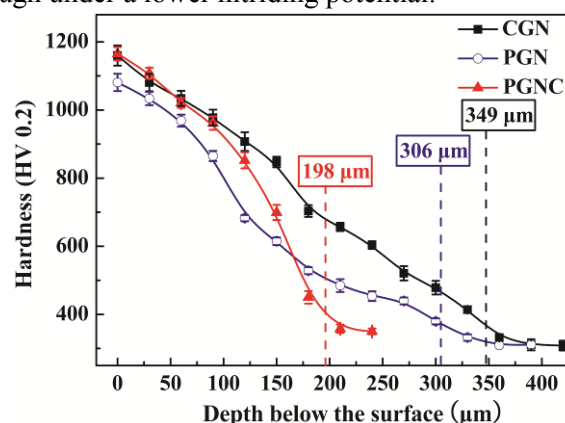
After the CR treatment to the PGN sample, a continuous flat surface without spalling was formed, and elongation of the microstructure along the rolling direction could be observed in the cross-sectional microstructure of the PGNC sample, as shown in Fig. 2a. The thickness of the plastic deformation nitrided layer is approximately 170  $\mu\text{m}$ . A TEM image and SAED pattern of the nitrides in the top surface layer of the PGNC sample are shown in Fig. 2b. Besides a high-density of dislocations, a few particles of about 100-200 nm sizes were also found in the matrix of the top surface layer. The corresponding electron diffraction pattern verified that these particles are  $\gamma'\text{-Fe}_4\text{N}$  nitrides.



**Figure 2.** (a) Cross-sectional microstructure of PGNC sample. (b) A TEM image and electron diffraction pattern (inset) of the nitrides in the top surface layer of the PGNC sample.

### 3.2. Hardness and toughness of the nitrided layer

Previous investigations have shown that the CL consisting of  $\epsilon$ -Fe<sub>2.3</sub>N and  $\gamma'$ -Fe<sub>4</sub>N phases presents a relatively high hardness with a high brittleness, whereas a thin mono-phase layer of  $\gamma'$ -Fe<sub>4</sub>N or a compound-free diffusion layer has a relatively low hardness with a good toughness and supports a high dynamical force [6,8,19]. Fig. 3 shows the hardness variation with depth below the surface of the steel 38CrMoAlA sample treated by different process. It was found that the surface microhardness of PGNC sample could reach about 1165 HV, which was notably higher than that of the PGN sample (1080 HV), and almost equal to that of the CGN sample (1160 HV). It was also evident that in the first 50  $\mu$ m of the nitrided layer, the hardness of the PGNC sample was higher than that of the CGN sample. This phenomenon can be explained by the dislocations and other defects induced by CR in the  $\alpha$  phase [11]. Based on the previous work, the effective case depth of the nitrided sample can be calculated as the case hardness value reaches the substrate hardness plus 50 HV [4]. The effective case depths are approximately 349, 306 and 198  $\mu$ m for the CGN sample, PGN sample and PGNC sample, respectively. The results demonstrated that the nitriding kinetic process could still be significantly accelerated by PGN, although under a lower nitriding potential.

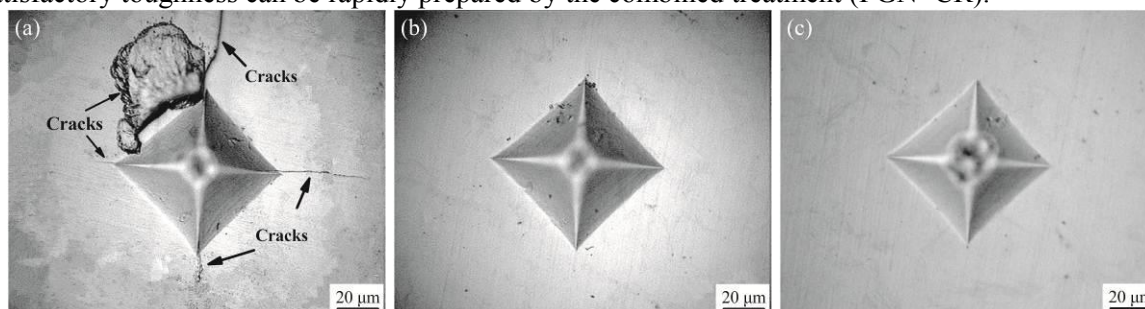


**Figure 3.** Variations in the microhardness along the depth from the top surface layer for the CGN sample, PGN sample and PGNC sample.

It has been shown that the morphological feature of the indentation produced by the hardness test reflects the bearing capacity and the toughness of the nitrided layers [4,20]. In order to qualitatively evaluate the toughness of the surface layer in each treated sample, Fig. 4 shows the indentation morphologies of the surfaces under a load of 10 kg. There are apparently different morphological features immediately noticed in these optical micrographs. It can be seen that, for the CGN sample (Fig. 4a), obvious linear cracks extending outward along each corner of the indentation could be noted,



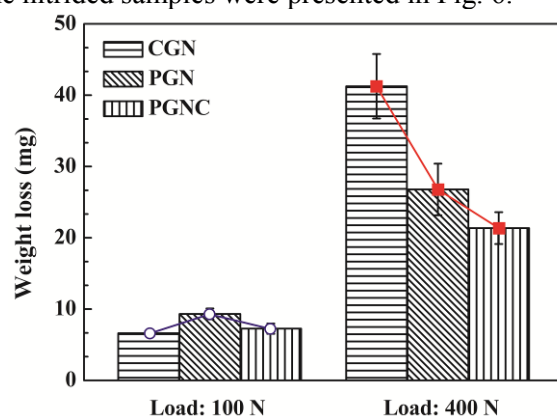
and serious damage near an edge was also observed. For comparison, there existed a regular indentation with no cracks in the PGN sample and PGNC sample after the test, as shown in Fig. 4b and c. The results indicated that the CGN sample presented the lowest bearing capacity and toughness. Hence, it can be confirmed that a compound-free nitrided layer with a relatively high hardness and satisfactory toughness can be rapidly prepared by the combined treatment (PGN+CR).



**Figure 4.** Optical micrographs of Vickers indentation for (a) CGN sample, (b) PGN sample and (c) PGNC sample under a load of 10 kg.

### 3.3. Wear properties

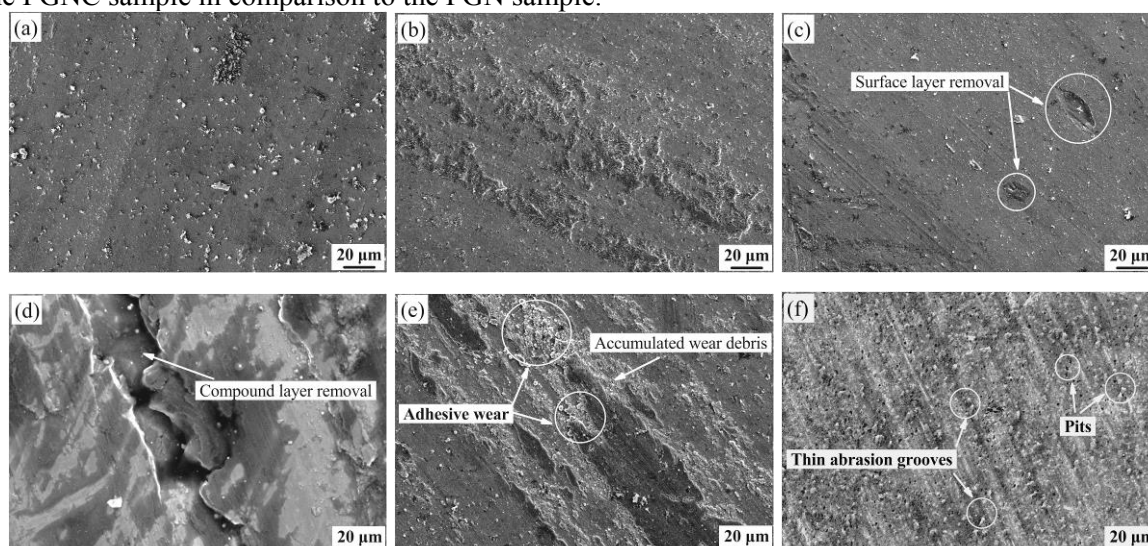
Fig. 5 shows the weight losses of wear scars after a wear test with a sliding distance of 170 m and at a load of 100 N and 400 N for the three treated samples, respectively. It can be seen that the wear weight loss measured at 100 N was significantly smaller than that obtained under the load of 400 N, but the corresponding wear behaviors were apparently different. It should be noted that the CGN sample, which exhibited the best wear resistance under the load of 100 N, had the largest wear loss amongst all the treated samples when the applied load was up to 400 N. Additionally, the wear loss of the PGNC sample was similar to one half of that of the CGN sample under the load of 400 N, indicating that excellent wear resistance could be obtained in the PGNC sample. The prior investigations have shown that the wear characteristics of the nitrided sample depend on many factors such as the compound layer composition, compound layer toughness, mode of mechanical loading, etc [5-8]. The compound layer will be flaked and formed into hard abrasive particles in the initial stage of sliding when the contact pressure exceeds a critical value [9]. These hard abrasive particles that stay in the contact zone between the pin and the disc will accelerate the abrasive wear and promote an increasing of the weight loss [5-7]. This would explain the wear behavior found for the nitrided samples in the present study. In order to further explain the wear characteristics occurring in each surface of the nitrided sample, the surface morphologies of the nitrided samples were presented in Fig. 6.



**Figure 5.** Wear weight loss for different samples at the applied loads of 100 N and 400 N.

Fig. 6a-c show the morphologies of the worn surfaces after a wear test with a load of 100 N and sliding distance of 170 m. Apparently, when the wear load was 100 N, no obvious traces of abrasive wear had been found in all of the sample surfaces. The worn surface of the CGN sample was very

smooth with some thin grooves (Fig. 6a), whereas the worn surface of the PGN sample was relatively rough with more grooves and spalling (Fig. 6b). The PGNC sample shared a similar wear mechanism with the CGN sample, and a small amount of surface layer removal was observed in this case (Fig. 6c). Increasing the wear load from 100 N to 400 N caused an obvious increase in the surface wear rate, as shown in Fig. 6d-f. Three different wear morphologies, indicative of different wear mechanisms, could be distinguished. The worn surface of the CGN sample is shown in Fig. 6d. It is clear that the compound layer was being removed from the wear track during testing, indicating that abrasive wear had occurred in this case. Traces of adhesive wear could be clearly observed on the worn surface of the PGN sample (Fig. 6e). The original surface had been worn away, and wear debris was agglomerated in the machined grooves and then formed the discontinuous compacted surface. The worn surface of the PGNC sample consisted of a large number of thin abrasion grooves and pits along the sliding direction, and no obvious surface layer removal was observed (Fig. 6f), demonstrating a minor weight loss during sliding. This emphasizes the fact that a good combination of hardness and toughness of the coating is crucial for good wear resistance [21]. The hardness distribution and compressive residual stress in the compound-free nitrided layer can be further improved by means of plastic deformation, which is attributed to the existence of a lot of defects [4,11]. Especially, the compressive residual stress is recognized to hinder the crack initiation and propagation [4,21]. Therefore, it is reasonable to believe that the wear resistance under heavy loads can be improved on the PGNC sample in comparison to the PGN sample.



**Figure 6.** SEM images of the wear scars formed at an applied load of 100 N in (a) CGN, (b) GN and (c) PGNC sample and at 400 N in (d) CGN, (e) PGN and (f) PGNC sample.

#### 4. Conclusions

A compound-free nitrided layer on steel 38CrMoAlA steel with a relatively low hardness but good toughness can be rapidly prepared by PGN with a controlled nitriding potential. After a CR treatment to the PGN sample, the surface hardness can be further improved without loss of toughness. The CGN sample treated for 30 h showed excellent wear resistance under light loads due to its higher surface hardness, but showed a serious deteriorated performance under heavy loads, which can be attributed to the fracture of the compound layer leading to the formation of the hard abrasive particles. The greatly enhanced hardness as well as the surface toughness contributed to the best bearing capacity and wear resistance under heavy loads in the combined treated sample (PGN for 5 h + 30% CR). The formation of the compound-free nitrided layer by using the PGN and CR technique not only provides a new approach to improve the surface hardness, toughness and wear resistance of the nitrided layer, but also increases the manufacturing efficiency.



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